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Cc:	Draper, Cynthia E; Bondy, Garret E; Fortenberry, Chase; Griffith, Garry T.; Abid, Joseph A; DeVisser, Anita
Subject:	Revised Area 3 SRI/FS Work Plan and Supplemental Field Sampling Plan - Response to Comments
Date:	Tuesday, July 09, 2013 12:55:05 PM
Attachments:	TestAmerica SOP subsampling.pdf

Provided below are responses to comments received via email from the MDEQ on June 27, 2013 for the Area 3 Field Sampling Plan. The responses are representative of our phone conference held July 2, 2013, discussion of the issues, agreements and conclusions. In addition, attached is the TestAmerica SOP for processing the incremental samples.

Incremental Sampling

1. One procedure had been discussed was conducting limited hand augers in the area of the incremental sampling to confirm the underlying stratigraphy. If we did not overlook the detail in the plan, MDEQ believes it is prudent to conduct some limited hand auguring in the areas of the incremental sampling to confirm the underlying stratigraphy of the sample material. Response: Bank sampling in the residential area will be conducted prior to the incremental sampling in the backyards. This approach will help AMEC to identify the "interval of interest" before conducting sampling in the backyards and give information on underlying stratigraphy that may be encountered in the backyards. AMEC recommends that any additional coring in the yards be conducted nearer to the river and not in every yard – especially not the well manicured lawns. MDEQ agrees that we should minimize augering in the yards, and if it becomes necessary to confirm the interval of interest, the boring would be conducted nearer to the river rather than closer to the house.

2. MDEQ notes that the residential areas may be areas of high heterogeneity. This is expected due to the distributional heterogeneity typical of floodplains and because the DUs proposed include both exposed (floodplain) and unexposed (upland backyard) "populations". The use of 30 increments is considered minimal and may result in higher than decided error, dependent upon our goals. It may be prudent to increase the number of increments to 49 (7 x 7 grid) to handle the anticipated heterogeneity. At this point it is not clear what number of increments is a best fit and will depend on heterogeneity of the area, sampling goals, and the proximity of the sample results to selected criteria. AMEC agrees that precision error would be reduced by increasing the number of increments. Based on the general rectangular shapes of the backyards, AMEC suggests a different grid shape (8 x 6). MDEQ agreed with this approach.

3. From a process standpoint we think it will be prudent to identify the sampling procedures before we get in the field. For example:
    a. Will both intervals be collected from one push of the probe or will intervals be collected separately.
    a. If collected together and full target depth of 12 inches is not recovered, identify how the aliquots are to be split into the 0-6' interval and 6-12' interval)
    b. The specific tool to be used is important (in order to predict the proper amount of sample mass).
    c. Identify a recovery minimum at which point resampling will be conducted.

AMEC tested the equipment, a soil push-probe (7/8" diameter) during site recon to evaluate the collection of the two intervals and recovery. The push probe appears to be appropriate for

collecting both intervals with good recovery. MDEQ is pleased with our equipment and field experience. The target is 75% recovery. Both AMEC and MDEQ acknowledge that site conditions may vary across the residential yards, and additional equipment (such as a spade-shovel) may be needed to collect samples at the predetermined intervals. MDEQ was concerned with potential cross contamination between 0-6" and 6-12". AMEC to follow up with research. AMEC and MDEQ agreed that if it appeared that there were apparent smearing (of gray material) between the two intervals, 2 push probe samplers would be a good solution, one to 6" and the other 6-12".

4. It is not clear how the eco assessment will be conducted. It appears that it will be conducted over an approximately 2000 foot stretch of the bank as one DU. Please clarify if multiple DUs will be sampled along the bank. Also, it is not clear if the 683 NGVD will be used as a hard line for eco sampling, or if the exact extent of the DU(s) will be based on field observations as well.

AMEC intends to establish the 683 elevation and use the area from this elevation to the water as the DU. However, given our observations during site recon, and topographic map information, there may not be much distance between the 683 elevation and the water, if any. As a backup plan, AMEC would mark off a strip 20 foot wide along the river, across contiguous residential properties. MDEQ agrees with this approach and backup plan. AMEC explained that goal is contiguous, but may be broken due to access issues. AMEC agreed to do triplicate in the ecological DU.

5. A critical component of IS is the laboratory processing of the IS sample after it is collected in the field. The specific lab and the specific lab processing procedures (including the specific subsampling, aliquot mass and analytical methods) will be important to identify before the sampling event.

AMEC plans to use TestAmerica Lab (Canton, Ohio) and their existing SOP. Disaggregation of the sample through crushing, and not grinding will prevent smearing. MDEQ agrees with the method. AMEC's contact at the lab is Mark Loeb. AMEC to provide laboratory SOP to MDEQ.

Bank Sampling

6. It appears from the FSP that different sample core processing is being proposed. MDEQ believes that retaining the previously approved processing regimen is best. For example, collecting cores with 3" Lexan tubes in lifts as appropriate, and dividing samples on the 0-6, 6-12, 12-24, etc. with segregation of material of interest. We understand that the floodplain in area 3 may require hand augering through the hard upper material, before proceeding with Lexan.

AMEC agrees with MDEQ intervals (0-6, 6-12 and 12-24) to be consistent with past sampling. Anita noted that the interval 12 to 24" may be saturated. MDEQ recommends sampling even if saturated. AMEC agreed to collect even if saturated. AMEC asked about specification of 3" vs. 2" diameter Lexan tubes. MDEQ recommends using the 3" diameter Lexan tubes due to their experience with less compression during advancement and better recovery. AMEC agreed to use the 3" Lexan tubes.

Mill race

7. The inclusion of 4 transects from the river edge, further upstream the former race to the parking lot area is appropriate. AMEC agreed to add another transect "upstream" in the old mill race and then evenly distribute the transects.
8. Make sure transects only occupy former race, as the line (in blue) depicts the race as being wider than aerials suggest. The width of the transects illustrated on Figure 4 may lie outside of the mill race channel. The MDEQ suggested that the transects be located in the field based on observations of topography and avoidance of bank soils. AMEC agrees with this method of transect placement.
9. Once all cores are collected along the transect, the Lexan should be observed and the most interesting core sent for analysis (as opposed to only selecting the middle core). Other cores sent to freezer pending results. Previous sampling results from the mill race area had highest concentrations of PCBs at the 681-682 elevation; AMEC believes it is important to reach this depth with advancement of hand auger and/or Lexan tubes. AMEC suggested that all samples collected along the transects be analyzed to provide more complete characterization of extent. The MDEQ agrees with this approach.
10. For all cores use traditional sectioning. Although AMEC had initially targeted the 681-682 elevation for sample collection, the method proposed by MDEQ (and previously employed) to collect samples at the 0-6", 6-12", 12-24" intervals and additional samples at intervals of interest within these predetermined intervals is an acceptable alternative. MDEQ noted that there were some sampling guidelines developed by the MDEQ to facilitate this type of sampling and they will provide these guidelines to AMEC.

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Title: SUBSAMPLING

[Method: ASTM D6323-98]

Approvals (Signature/Date):

Technology Director	Date 5/28/09	Health & Safety Coordinator	Date 6/01/09
Quality Assurance Manager	Date 6/1/09	Laboratory Director	Date 6/1/09

This SOP was previously identified as SOP No. NC-IP-001, Rev 6, dated 05/28/08

Facility Distribution No. _____ **Distributed To:** _____

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. These mixing, particle size reduction and subaliquoting techniques are applicable to a wide range soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, analyte and quality objectives.

1.2. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. Air-Dried Soil Mixing Process--the sample is air dried at room temperature and chopped or ground to a fine powder to facilitate obtaining a representative sub-sample and improving analyte extraction efficiency.

2.2. Soil Fractionation for Lead--applicable to lead impacted soil and is designed to distinguish the lead in the fine soil fraction from the coarse soil fraction.

2.3. Multi-Increment Sample Wet Mixing Process--applicable to mixing multi-increment samples into a single composite. These samples are NOT air dried, but rather water is added to facilitate mixing in a heavy-duty mixer.

2.4. Solid sample mixing procedures include in-jar mixing, horizontal surface mixing, and mortar and pestle.

2.5. Solid sample subaliquoting procedures include alternate scoop, line and scoop, and cone and quarter methods.

2.6. Liquid sample subaliquoting procedures include centrifuge, pipettes, coliwasa devices, and multiphase procedures.

3. DEFINITIONS

3.1. Refer to the glossary in the TestAmerica North Canton Quality Assurance Manual (QAM), current version.

3.2. Mix – To thoroughly mix the sample and reduce the analyte concentration differences between different parts of the overall sample.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.4. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.

5.5. When operating the electric chopper or grinder, be sure to keep all liquids clear to prevent the risk of electrical shock from any spills.

5.6. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection.

6. EQUIPMENT AND SUPPLIES

6.1. Drying tray, plastic or aluminum

6.1.1. Half cake sheet pan, Pactiv #614255, or equivalent

6.1.2. Quarter sheet cake pan, Pactiv #604245, or equivalent

6.2. Butcher Paper

6.3. Plastic wrap

6.4. Mortar and pestle

6.5. Food chopper, Black and Decker Handi Chopper or DeLonghi Mini Food Processor or equivalent

6.6. Coffee grinder, Kitchen Aid BCG100 or equivalent

6.7. Wooden spatula, 6 in.

6.8. Stainless steel sieves, 1 mm, #10, #20, #60, ¼ inch

6.9. Electrolux Assistant 8 qt mixer, with dough hook

6.10. Large cookie scoop, with hand actuated ejector blade

6.11. Small cookie scoop, with hand actuated ejector blade

6.12. Fluoropolymer scoop, not commercially available

6.12.1. Construct by fastening the bottom half of a fluorpolymer bottle to a wooden handle with stainless steel screws.

6.13. Stainless steel pot with cover, Bain-Marie 6 qt.

6.14. Wooden spoon, 12" long

Picture of labware for multi-increment sample wet mixing process (Section 11.3.2.5) - large scoop, small scoop, wooden spoon, fluoropolymer scoop, stainless steel pot/cover

7. REAGENTS AND STANDARDS

7.1. Deionized water – Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Not applicable to this procedure. Sample collection, preservation, and storage is dependent on the requested test method.

9. QUALITY CONTROL

9.1. Not applicable to this procedure

10. CALIBRATION AND STANDARDIZATION

10.1. Not applicable to this procedure

11. PROCEDURE

11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. The following sections describe a variety of procedures. Select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives.

11.3.2. Solid Sample Mixing and Particle Size Reduction Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wood blade.

11.3.2.3. Mortar and pestle

11.3.2.3.1. A mortar and pestle can be used for samples in the 1 to 8 mm particle size. The final reduced size is between 5um and 8 mm. This process can be used for wet or dry, organic or inorganic substances.

11.3.2.3.2. Fill the mortar about 1/3 with sample. Grind and mix the sample with the pestle. Transfer the processed sample to a separate container, and repeat the grind-and-mix step with additional sample aliquots as needed.

11.3.2.4. Dry mixing (multi-increment sampling support)

11.3.2.4.1. Air dry, chop, sieve, mix

11.3.2.4.1.1. Line a standard aluminum tray with a disposable aluminum tray ($^1/_2$ or $^1/_4$ sheet depending on sample size). If aluminum is a metal of interest line the tray with butcher paper.

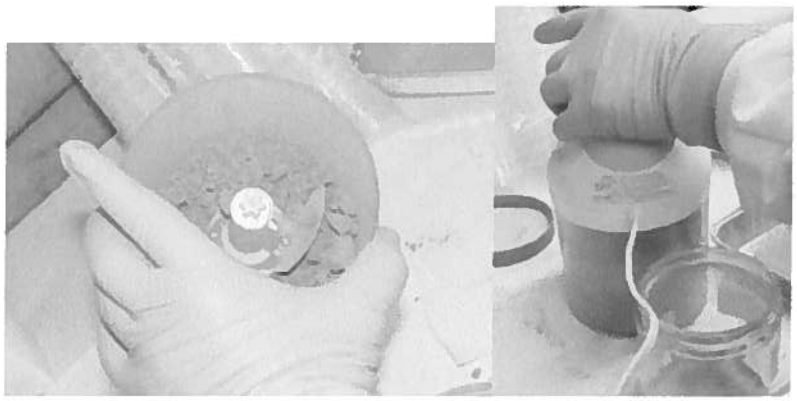
11.3.2.4.1.2. Remove large rocks and vegetation. Do not decant the free water. Mix the sample and remove approximately 10 g for total solids analysis. Spread all remaining sample in a thin layer in the tray.

11.3.2.4.1.3. Place the tray in the ventilated drying rack for up to five days at room temperature.

11.3.2.4.1.4. Periodically, stir the sample to expose moist sample to the air.

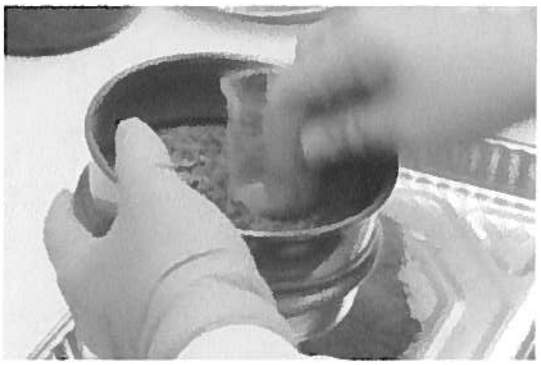
11.3.2.4.1.5. Allow sediment samples to dry to less than 30% moisture content. Soil samples should be less than 15% moisture content. The dried sample must be crushable, and not prone to sticking together.

11.3.2.4.1.6. Pulverize the dried sample to break up the dried sample clumps with a bladed chopper, but do not grind the small pebbles into powder.



11.3.2.4.1.7. If the volume capacity of the chopping equipment is large enough, transfer the whole sample to the chopper. If sample volume is too large, pulverize the sample in sub-aliquots. Combine all pulverized sub-aliquots, and mix thoroughly by stirring, shaking or tumbling.

11.3.2.4.1.8. Pass the sample through a sieve to remove small pebbles and organic materials. Project specific guidelines will determine the sieve opening size. Typically, a #10 sieve is used for soils and a #20 sieve for sediments.



11.3.2.4.2. Air dry, chop, mix

11.3.2.4.2.1. When the project objectives dictate that all small and medium sized materials be included in the final sample, use the procedure described in Section 11.3.2.4.1, but skip the sieving step in Section 11.3.2.4.1.8.

11.3.2.4.2.2. Collecting a representative subaliquot is more difficult when the sample has a variety of particle sizes. Refer to the subaliquoting section below for recommended procedures. The line and scoop process in Section 11.3.3.4 is particularly applicable to dry flowable samples with a wide range of particle sizes.

11.3.2.4.3. Other

11.3.2.4.3.1. Various permutations of soil chopping and sieving are possible depending on the needs of the project and can be accommodated but must be clearly defined in consultation with the client. These variations can include different sieve sizes and changing the order so that sieving is

done prior to chopping. This would most likely be done to exclude a particular size of organic or rocky material.

11.3.2.5. Wet mixing (multi-increment sampling support)

11.3.2.5.1. Weigh 100.0 +/- 0.2 g of each ¼ acre sub-sample. Stir the sample prior to and during the transfer from the original container to the weighing container. Record the weight to two decimal places.

11.3.2.5.1.1. If there are fewer than 40 sub-samples, increase the weight of sample proportionally. Calculate the new target weight as follows: target weight = 4000g/# sub-samples.

11.3.2.5.1.2. Exclude rocks, organic matter, and other debris from the weighed sub-sample by sieving through a ¼ inch sieve prior to weighing when such material is present.

11.3.2.5.1.3. The sieve may also be used to break up clay chunks. Sieving may be done either before or after weighing for this purpose. Use only when needed. Hard dry soil agglomerates should be broken up by hand crushing or chopping with a food chopper.

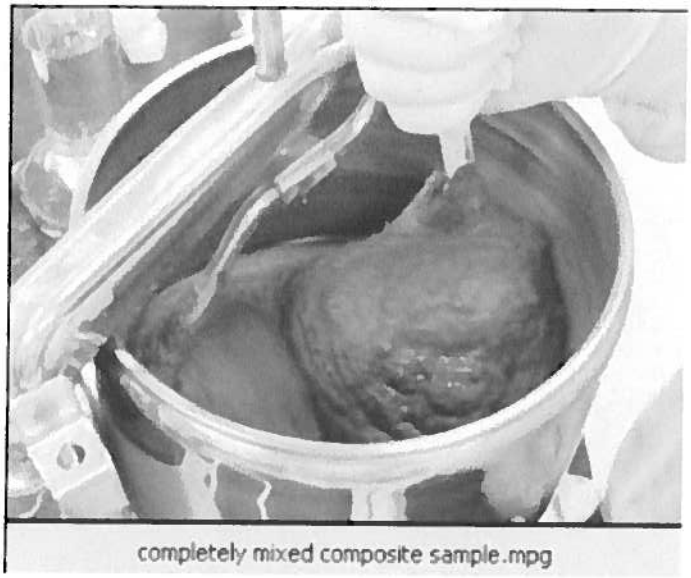
11.3.2.5.1.4. If a sub-sample is over ½ gravel, expand the maximum particle size from ¼ inch to 1.8 inches.

11.3.2.5.2. Transfer the weighed (and sieved as needed) sub-sample aliquot to a stainless steel pot used to collect all weighed aliquots for that composite sample. The total mass of the composite sample will be at least 4 kg.

11.3.2.5.3. Repeat Section 11.3.2.5.1 until all sub-samples have been weighed and transferred to the compositing pot.

11.3.2.5.4. Assemble the heavy-duty mixer with mixing hook. The mixing hook must be inserted high in the mounting bracket to avoid dragging on the bottom of the bowl and allow small stones to pass under the hook. Transfer the entire composite sample from the covered stainless steel pot to the stainless mixing bowl.

11.3.2.5.5. Turn on mixer. Add reagent water to ensure complete mixing of the sample. The mixed sample will have uniformly distributed water, look visually homogeneous, and have the consistency of a thick paste. Do not add so much water as to form runny slurry. Use a wooden spoon to assist the mixing process by scraping mud from the sides and directing it to the center. The picture below shows the proper consistency. Mix for three minutes after the proper consistency has been achieved.



completely mixed composite sample.mpg

11.3.2.5.6. Record the volume of reagent water added and the total mixing time.

11.3.2.5.7. Scrape the mud from the wooden spoon and mixing hook.

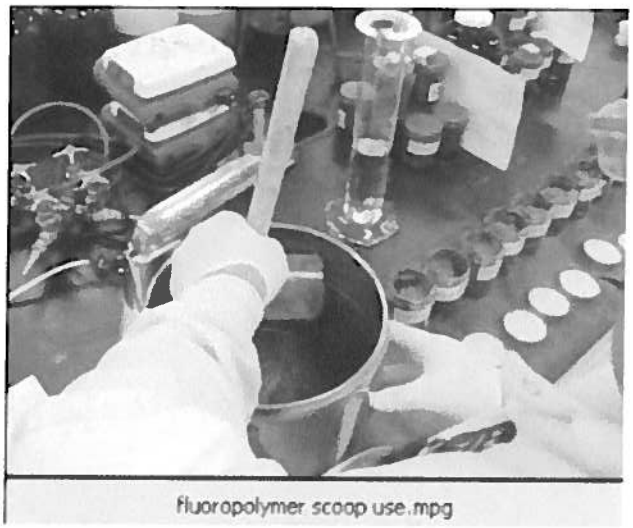
11.3.2.5.8. Split the composite sample between 5-15 250 mL jars, depending on project requirements.

11.3.2.5.9. Use the large scoop to dispense the equal aliquots of the composite sample--one aliquot into each of 8 oz. jars. Repeat the process if there is sufficient sample for a complete set of large scoop aliquots (per Section 11.3.2.5.8).

large scoop use.mpg

11.3.2.5.10. Once the volume of wet composite sample in the bowl is less than the determined set of large scoops, switch to aliquoting with the small scoop. Dispense an equal amount of small scoops--one into each 8 oz. jar.

11.3.2.5.11. Use the fluoropolymer scoop to scrape inside the bowl, and dispense this part of the sample with the small scoop. If there is insufficient sample to use full scoops, the use of replicate partial scoops is acceptable.



fluoropolymer scoop use.mpg

11.3.2.5.12. When aliquoting is complete, the composite sample has been evenly distributed among the 8 oz. jars. Note: Multiple jars are used for various analyses, quality control, and archive purposes.

11.3.2.5.13. Mix the contents of each 250 mL jar, and remove about 10 grams from the each of the first three jars for three total solids (percent moisture) analyses. The relative percent difference (RPD) between the first two must not exceed 8%.

11.3.2.5.13.1. If the RPD exceeds 8%, repeat the total solids analysis on two fresh aliquots from the first two jars.

11.3.2.5.13.2. If the RPD still exceeds 8%, repeat the mixing process in Sections 11.3.2.5.1 to 11.3.2.5.12

11.3.2.5.14. Wipe the top of the jar to remove excess sample and install the cap. Transport all sample containers to Sample Receiving.

11.3.2.5.15. Discard the wooden spoon and spatulas. Wash the mixer bowl, hook and scoops using soap and water. Rinse with tap and reagent water.

11.3.3. Solid Sample Subaliquoting Procedures

11.3.3.1. Multiple increments from a jar

11.3.3.1.1. Select 5-10 small sample increments from various locations within the sample container to make the entire subaliquot. The increments must come from all general areas of the container--top, sides, center, and bottom.

11.3.3.2. Multiple increments from a "pancake" spread sample

11.3.3.2.1. Spread the sample to a consistent depth on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum foil or plastic depending on the analytes of interest).

11.3.3.2.2. Select 30 or more small increments spread evenly over the sample area.

11.3.3.2.3. If the sample has noxious odors or produces dust, the sample spreading and increment collecting should be performed in a hood or large flat bag of appropriate material. The analyst must be protected from potential inhalation hazards from the sample.

11.3.3.3. Alternate scoop

11.3.3.3.1. Wet or dry solid samples can be divided into two or more smaller aliquots. Determine the number of subaliquots and prepare that many empty containers

11.3.3.3.2. Scoops of the mixed laboratory sample are either placed in the analytical vessel or discarded.

11.3.3.3.3. Three scoops are discarded for every scoop saved. Randomly select an aliquot and place in the first container. Place the next three aliquots in the second container. Repeat as needed for additional aliquot containers.

11.3.3.3.4. Repeat the aliquoting cycle until the original sample is consumed.

11.3.3.4. Line and scoop

11.3.3.4.1. This process is intended to produce large sub-samples from very large dry flowable solid samples, such as those collected using field multi-increment sampling, dried and then chopped using the process described in Section 11.3.2.4. See Reference16.1.7.

11.3.3.4.2. Pour the dry soil sample into a long thin pile onto a clean horizontal surface. The pour height should not exceed 20 cm to minimize the formation of a dust cloud.

11.3.3.4.3. Ensure that the sample container makes at least 20 passes back and forth over the "line of sample".

11.3.3.4.4. Using a rectangular flat-bottomed scoop remove an increment from the "line of sample". Ensure that a complete cross cut of the sample line includes the entire depth of that increment. Combine increments as needed to produce the needed sub-sample size.

11.3.3.5. Cone and quarter (for dry flowable samples)

11.3.3.5.1. Pour the sample into a cone on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum foil, or plastic depending on the analytes of interest).

11.3.3.5.2. Cut the cone in half in two directions to form four quadrants.

11.3.3.5.3. Return opposite quadrants to the original sample container.

11.3.3.5.4. Repeat the process in Sections 11.3.3.5.1 through 11.3.3.5.3 until the needed aliquot size has been obtained.

11.3.3.6. Soil fractionation for lead analysis

11.3.3.6.1. Add sample ID label to outside of a disposable aluminum ($^1/_2$ or $^1/_4$ sheet depending on sample size). Line with butcher paper if aluminum is also an analyte of interest.

11.3.3.6.2. Transfer the entire soil sample to a tray. Remove large rocks and vegetation. Mix the sample, and remove approximately 10g for total solids analysis. Spread all remaining sample in the tray. Spreading the sample in a thin layer speeds drying and reduces the formation of hard clay chunks.

11.3.3.6.3. Place the tray in the ventilated drying rack for up to five days.

11.3.3.6.4. Periodically, stir the sample if needed to expose moist sample to the air.

11.3.3.6.5. Wet samples with high clay content tend to form large hard clay chunks. To reduce the formation of these hard "bricks", use the bottom of a clean disposable beaker to gently crush the semi-dried sample before drying is complete.

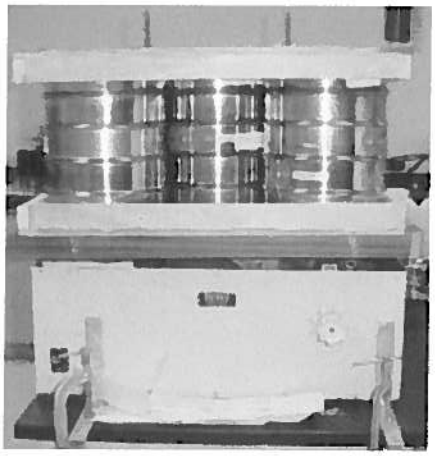
11.3.3.6.6. Allow the sample to dry until crumbly.

11.3.3.6.7. Assemble 8-inch sieve stack. The order from the bottom of the stack is collection pan, #60 sieve, and #10 sieve. Transfer sample ID label from drying pan to sieve stack.

11.3.3.6.8. Transfer dried soil to the #10 sieve at the top of the sieve stack. If fine particulates are present, use sufficient ventilation to prevent the analyst from inhaling the dust. Install sieve cover.

11.3.3.6.9. Some wet clay samples will dry into a hard "brick" or "pancake". Break by hand until the chunks are small enough to fit into the top sieve and proceed. This brick-forming tendency can be reduced by gently breaking the semi-moist chunks during the drying process of Section 11.3.3.6.4.

11.3.3.6.10. Place the sieve stack on the shaker table and secure. Run the shaker on high until most of the sample has passed through the #10 sieve (10 minutes to 2 hours depending on sample volume and size of soil agglomerates). The high shaker setting produces approximately 180 cycles/min with 4 cm travel on the back and forth shaking.



11.3.3.6.11. Note: Some hard clay samples may have a large percentage of soil agglomerates that do not crumble and pass through the #10 sieve. Do not extend shaking beyond two hours without conferring with the client.

11.3.3.6.12. Record the weights of the three fractions--large (did not pass through the #10 sieve), coarse (passed through #10 sieve, but not the #60 sieve), and fine (passed through both #10 and #60 sieves).

11.3.3.6.13. To weigh each fraction, place the corresponding sieve or pan on the balance, press "Tare", transfer the fraction, and place sieve or pan back on the balance. The weight of the fraction will read as a negative value.

11.3.3.6.14. Discard the large fraction. Mix the fine fraction. Remove approximately 10g for total solids analysis.

11.3.3.6.15. If the coarse fraction is to be analyzed separately, mix the coarse fraction and remove approximately 10g for total solids analysis. The coarse fraction should be ground in a mortar and pestle (Section 11.3.2.3) prior to removing the aliquot for metals digestion.

11.3.3.6.16. The combination of coarse and fine fractions is defined as the "total". If the "total" is to be measured, weigh 1/10 of the coarse fraction and 1/10 of the fine fraction. Combine, mix, and remove approximately 10g for total solids analysis.

11.3.3.6.17. Bottle coarse, fine, and "total"(if needed) fractions for metals analysis.

11.3.3.6.18. For Michigan samples, the "total" result is calculated from a weighted average of the results from the fine and coarse fractions.

$$\text{Total Lead} = [(A \times Wf) + (B \times Wc)] / (Wf + Wc)$$

Where:
A = Concentration of Lead (mg/Kg dry) in fine fraction
B = Concentration of Lead (mg/Kg dry) in coarse fraction
Wf = Total weight of fine fraction
Wc = Total weight of coarse fraction

11.3.3.6.19. Wash the sieves with soap, tap water, and deionized water. Dislodge objects from the screen with a green scratch pad, wooden tongue blade or small screwdriver, as necessary. Dry sieves in a heated oven or air-dry at ambient temperature over night, depending how soon they will be needed.

11.3.4. Liquid Sample Mixing Procedures

11.3.4.1. Closed container shaking

11.3.4.1.1. All liquid samples should be mixed by shaking in the original closed sample container unless a multiple layer subaliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be subaliquoted using an appropriate technique as described below.

11.3.4.1.2. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.5. Liquid Sample Subaliquoting Procedures

11.3.5.1. Pour

11.3.5.1.1. Samples that are well mixed can be subaliquoted by pouring an appropriate volume off the top of the sample.

11.3.5.2. Layer Subaliquoting

11.3.5.2.1. Some liquid samples with multiple layers separate too quickly to pour a representative subaliquot off the top. In some instances it is best to separate the layers and handle them as individual samples. In other instances representative aliquots of each layer must be collected and processed as a single subsample.

11.3.5.2.2. Unless directed otherwise, record the phase ratios (or volumes) of the layers.

11.3.5.2.3. Gravity or Centrifuge Settling

11.3.5.2.3.1. Allow the sample layers to separate based on density. Centrifugation can be used to accelerate the process if simple gravity settling is not fast enough.

11.3.5.2.4. Separatory Funnel

11.3.5.2.4.1. Gently pour the sample into a separatory funnel. Allow time for additional layer separation as needed.

11.3.5.2.4.2. Drain the layers out into separate containers one at a time.

11.3.5.2.4.3. Some oils stick to the separatory funnel sides so the draining process must be slow enough to avoid remixing the layers.

11.3.5.2.5. Pipette

11.3.5.2.5.1. Use a pipette of appropriate size to collect a subaliquot of a sample layer of interest and transfer to an empty container.

11.3.5.2.5.2. If the entire sample is to be separated, pipette almost all the top layer into a new container. Draw the small volume of the top layer into the pipette along with a small portion of the second layer. Allow the two layers to separate in the pipette (like a separatory funnel). Dispense the bottom layer back into original sample container with the bulk of the bottom layer. Next dispense the remaining top layer into the container that holds the top layer.

11.3.5.2.5.3. Repeat as need for each layer of interest.

11.3.5.2.6. Coliwasa

11.3.5.2.6.1. A coliwasa is a "coring device" designed for liquid multilayer samples. It is a long tube with a short valve at the bottom. When designed and used properly it collects representative aliquots of each layer with each subaliquoting attempt.

11.3.5.2.6.2. The volume collected is determined by the diameter of the coliwasa and the height of the sample. The larger the tube diameter or taller the sample height the large the volume of sample collected.

11.3.5.2.6.3. Place the foot of the valve and its control rod in the multi-layer sample. Slowly slide the coliwasa tube over the control rod and close the ground glass seal of the valve at the bottom.

11.3.5.2.6.4. Lift the coliwasa by the control rod to keep the valve sealed.

11.3.5.2.6.5. Place the coliwasa over the new sample aliquot container. Grasp the top of the coliwasa tube and slowly lower the control rod a few millimeters to open the foot valve and drain the sample into the container.

11.3.5.2.6.6. Repeat Sections 11.3.5.2.6.3 through 11.3.5.2.6.5 until sufficient sample aliquot has been collected.

11.3.6. Multiphase Sample Mixing Procedures

11.3.6.1. All liquid samples should be mixed by shaking unless a multiple layer subaliquoting procedure is used. This applies emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be subaliquoted using an appropriate technique as described below.

11.3.6.2. Closed container shaking

11.3.6.2.1. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.6.3. Open container stirring

11.3.6.3.1. The sample is stirred or swirled to mix.

11.3.6.4. Wet Sediments

11.3.6.4.1. Aqueous solutions for non-volatile compounds may contain settleable materials. If the settleable materials are to be included as part of the laboratory sample, and they will remain suspended, or can easily be re-suspended and will remain so during the subsampling operation, the sample should be handled as a liquid sample. These samples should be gently swirled for 15 seconds or slowly inverted six times to reduce heterogeneity.

11.3.6.4.2. If the liquid portion only is to be used, the settleable material must be allowed to sink to the bottom before withdrawing the subsample.

11.3.6.4.3. If the settleable material will not remain suspended and is to be included in the analysis, the sample should be treated as a multilayered sample.

11.3.7. Multiphase Sample Subaliquoting Procedures

11.3.7.1. Multilayered samples may include liquid/liquid layers, liquid/solid samples, or solid/solid samples.

11.3.7.2. If the liquid portion of a sludge can be re-mixed with the solid portion and will re-separate over time, the sample should be handled as a solid sample.

11.3.7.3. If the solid portion will remain suspended or can easily be re-suspended, the sample should be treated as a liquid sample. Mixing may occur by inverting the container or with slight shaking

11.3.7.4. Layer Separating

11.3.7.4.1. Gravity/Centrifuge – The solid/liquid phase separation may be achieved by gently centrifuging the unopened container or by allowing it to sit undisturbed until the solid portion is settled.

11.3.7.4.2. Pipette – For liquid/liquid layers, separate the layers using a pipette. Each layer may be either transferred into another container or directly into the analytical vessel. Each separated portion is then handled as a homogeneous liquid sample.

11.3.7.4.3. Filter/Decant – For liquid/solid layers, the liquid subsample may be obtained by filtering or decanting the liquid portion from the solid portion. The liquid portion is then handled as a homogeneous liquid sample. The solid portion is handled as a solid laboratory sample.

11.4. Sample Analysis

11.4.1. Not applicable to this procedure

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, which may be in an electronic format, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

13. METHOD PERFORMANCE

13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste management and Pollution Prevention".

15. WASTE MANAGEMENT

15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.2. Waste Streams Produced by the Method

15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. U.S. EPA, 2000. TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites. EPA-540-F-00-010. OSWER 9285.7-38. April. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/sssiev.pdf

16.1.2. U.S. EPA, 2003. TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges. OSWER 9285.7-37. March. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/firing.pdf

16.1.3. U.S. EPA, 2003. Superfund Lead-Contaminated Residential Sites handbook. OSWER 9285.7-50. August. Available on-line at: http://www.epa.gov/superfund/programs/lead/products/handbook.pdf

16.1.4. Michigan DEQ SOP #213 Revision #1, Nov. 9, 2004, Soil Fractions Preparation for Lead Analysis (Creating Total, Fine and Coarse Soil Samples). Available on-line at: http://www.deq.state.mi.us/documents/deq-rrd-OpMemo_2_SoilFractionsPrepForLead.pdf

16.1.5. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.6. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.1.7. Draft ASTM Standard, by Bill Ingersoll, Soil Subsampling For Explosive Residue, Section 14.6 First Stage Material Delimitation and Extraction: Multiple Increment Scoop Subsampling

16.1.8. ASTM D 6323-98, Laboratory Subsampling of Media Related to Waste Management Activities

16.1.9. Corporate Quality Management Plan (CQMP), current version

16.1.10. Revision History

Historical File:		Revision 1: 05/19/99		Revision 5: 11/06/06
		Revision 2: 02/23/04		Revision 6: 05/28/08
		Revision 3: 05/24/04		
		Revision 4: 10/13/06		

16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Supplemental Practices for DoD Project Work, NC-QA-016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Reporting limits

17.1.1. Not applicable to this procedure

17.2. Method deviations – None